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(54) METHOD FOR PRODUCING SILICATE PHOSPHOR

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a method for producing a silicate phosphor having high luminance and to provide the phosphor produced by the method for production.

SOLUTION: The method for producing the silicate phosphor comprises firing a mixture of metal compounds. In the method, a spherical silicon oxide is used as one of the metal compounds. The silicate phosphor comprises a compound represented by general formula $mM1O-nM2O-2SiO_2$ (wherein, M1 is one or more selected from the group consisting of Ca, Sr and Ba; M2 is one or more selected from the group consisting of Mg and Zn; m is ≥ 0.5 and ≤ 3.5 ; and n is ≥ 0.5 and ≤ 2.5) and one or more selected from the group consisting of Eu and Mn as an activator. The silicate phosphor is obtained by the method for production described in any thereof and contains ≥ 80 wt.% of particles having $\leq 5 \mu m$ primary particle diameter.

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最終頁に続く

(54) 【発明の名称】 ケイ酸塩蛍光体の製造方法

(57) 【要約】

【課題】輝度が高いケイ酸塩蛍光体の製造方法およびその製造方法により製造された蛍光体を提供する。

【解決手段】金属化合物の混合物を焼成することによるケイ酸塩蛍光体の製造方法において、球状の酸化ケイ素を金属化合物の一つとして用いるケイ酸塩蛍光体の製造方法。ケイ酸塩蛍光体が、一般式 $mM^1O \cdot nM^2O \cdot 2SiO_2$ (式中の M^1 はCa、SrおよびBaからなる群より選ばれる1種以上、 M^2 はMgおよびZnからなる群より選ばれる1種以上、mは0.5以上3.5以下、nは0.5以上2.5以下である。)により表される化合物と、付活剤としてEu、Mnからなる群より選ばれる1種以上とを含んでなるケイ酸塩蛍光体である上記記載の製造方法。上記のいずれかに記載の製造方法によって得られ、一次粒子径が5 μm以下の粒子が80重量%以上含まれるケイ酸塩蛍光体。

(2)

【特許請求の範囲】

【請求項1】

金属化合物の混合物を焼成することによるケイ酸塩蛍光体の製造方法において、球状の酸化ケイ素を金属化合物の一つとして用いることを特徴とするケイ酸塩蛍光体の製造方法。

【請求項2】

球状の酸化ケイ素の長径／短径比が1以上1.5以下である請求項1記載の製造方法。

【請求項3】

球状の酸化ケイ素の平均粒径が0.1 μm 以上3 μm 以下である請求項1または2に記載の製造方法。

【請求項4】

球状の酸化ケイ素のBET比表面積が $1\text{m}^2/\text{g}$ 以上 $30\text{m}^2/\text{g}$ 以下である請求項1～3のいずれかに記載の製造方法。

【請求項5】

金属化合物の混合物が、Ca、Sr、Ba、Mg、Eu、MnおよびZnからなる群より選ばれる一種以上の金属元素の化合物とSiの化合物の混合物である請求項1～4のいずれかに記載の製造方法。

【請求項6】

焼成における最高到達温度が 1000°C 以上 1400°C 以下の温度範囲である請求項1～5のいずれかに記載の製造方法。

【請求項7】

ケイ酸塩蛍光体が、一般式 $m\text{M}^1\text{O} \cdot n\text{M}^2\text{O} \cdot 2\text{SiO}_2$ （式中の M^1 はCa、SrおよびBaからなる群より選ばれる1種以上、 M^2 はMgおよびZnからなる群より選ばれる1種以上、 m は0.5以上3.5以下、 n は0.5以上2.5以下である。）により表される化合物と、付活剤としてEu、Mnからなる群より選ばれる1種以上とを含んでなるケイ酸塩蛍光体である請求項1～6のいずれかに記載の製造方法。

【請求項8】

請求項1～7のいずれかに記載の製造方法によって得られることを特徴とするケイ酸塩蛍光体。

【請求項9】

一次粒子径が $5\mu\text{m}$ 以下の粒子が80重量%以上含まれる請求項8記載のケイ酸塩蛍光体。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】

本発明は、ケイ酸塩蛍光体の製造方法に関し、特にプラズマディスプレイパネル（PDP）、希ガスランプなどの真空紫外線励起発光素子用に適したケイ酸塩蛍光体の製造方法に関する。

【0002】

【従来の技術】

ケイ酸塩蛍光体は、蛍光灯、ブラウン管、蓄光体、真空紫外線励起発光素子等に用いられている。特にPDPなどの真空紫外線励起発光素子においては、素子の輝度向上が強く望まれており、それらに用いられるケイ酸塩蛍光体の輝度向上が求められている。

【0003】

ケイ酸塩蛍光体は、従来から金属化合物の混合物を焼成することにより製造されている。ケイ素源となる金属化合物としては酸化ケイ素が用いられており、従来の製造方法においては、粒子形状が不定形のものが、工業的に入手容易で安価であるため用いられていた。

【0004】

【発明が解決しようとする課題】

本発明の目的は、輝度が高いケイ酸塩蛍光体の製造方法およびその製造方法により製造された蛍光体を提供することにある。

(3)

【0005】

【課題を解決するための手段】

本発明者らは、かかる状況下、鋭意研究を重ねた結果、金属化合物の混合物を焼成することによるケイ酸塩蛍光体の製造方法において、原料となる酸化ケイ素として球状の酸化ケイ素を金属化合物の一つとして用いると、発光の輝度が高いケイ酸塩蛍光体が製造できることを見出し、本発明を完成するに至った。

【0006】

すなわち本発明は、金属化合物の混合物を焼成することによるケイ酸塩蛍光体の製造方法において、球状の酸化ケイ素を金属化合物の一つとして用いるケイ酸塩蛍光体の製造方法を提供する。また本発明は、球状の酸化ケイ素の長径／短径比が1以上1.5以下である前記製造方法を提供する。また本発明は、球状の酸化ケイ素の平均粒径が0.1 μm 以上3 μm 以下である前記いずれかに記載の製造方法を提供する。また本発明は、球状の酸化ケイ素のBET比表面積が1 m^2/g 以上30 m^2/g 以下である前記いずれかに記載の製造方法を提供する。また本発明は、金属化合物の混合物が、Ca、Sr、Ba、Mg、Eu、MnおよびZnからなる群より選ばれる一種以上の金属元素の化合物とSiの化合物の混合物である前記いずれかに記載の製造方法を提供する。また本発明は、焼成における最高到達温度が1000℃以上1400℃以下の温度範囲である前記いずれかに記載の製造方法を提供する。また本発明は、ケイ酸塩蛍光体が、一般式 $\text{mM}^1\text{O} \cdot \text{nM}^2\text{O} \cdot 2\text{SiO}_2$ （式中の M^1 はCa、SrおよびBaからなる群より選ばれる1種以上、 M^2 はMgおよびZnからなる群より選ばれる1種以上、mは0.5以上3.5以下、nは0.5以上2.5以下である。）により表される化合物と、付活剤としてEu、Mnからなる群より選ばれる1種以上とを含んでなるケイ酸塩蛍光体である前記いずれかに記載の製造方法を提供する。また本発明は、前記いずれかに記載の製造方法によって得られるケイ酸塩蛍光体を提供する。さらに本発明は、一次粒子径が5 μm 以下の粒子が80重量%以上含まれる前記記載のケイ酸塩蛍光体を提供する。

【0007】

【発明の実施の形態】

以下に本発明について詳しく説明する。

本発明のケイ酸塩蛍光体の製造方法は、金属化合物の混合物であって焼成によりケイ酸塩蛍光体を構成しうる混合物を焼成するケイ酸塩蛍光体の製造方法において、金属化合物の一つとして、球状の酸化ケイ素を用いる。前記金属化合物には、付活剤である金属元素の化合物も含まれる。本発明の製造方法においては、酸化ケイ素は球状の粒子からなる粉末である。球状の酸化ケイ素を用いることにより、理由は明らかではないが、輝度が高い蛍光体得られるのである。

【0008】

ここで、酸化ケイ素の粒子としては、電子顕微鏡写真を用いて計測した場合において1個の一次粒子の直径のうちで最も大きなものを長径、最も小さなものを短径とした場合、長径／短径比は1.5以下が好ましく、1.3以下がさらに好ましい。なお、酸化ケイ素の粒子が完全な球であれば、長径／短径比は1となる。酸化ケイ素の平均粒径は0.1 μm 以上3 μm 以下が好ましく、より好ましくは0.2 μm 以上2 μm 以下であり、さらに好ましくは0.2 μm 以上1.7 μm 以下である。球状の酸化ケイ素のBET比表面積は1 m^2/g 以上30 m^2/g 以下が好ましく、さらに好ましくは2 m^2/g 以上20 m^2/g 以下である。球状の酸化ケイ素の長径／短径比が1.5を超える場合、平均粒径が0.1 μm 未満または3 μm を超える場合、BET比表面積が1 m^2/g 未満または30 m^2/g を超える場合は、高い輝度が得られないおそれがある。

【0009】

本発明の製造方法において、金属化合物の混合物で焼成によりケイ酸塩蛍光体を構成しうる混合物に含まれる金属化合物で、酸化ケイ素以外の化合物としては、金属元素の水酸化物、炭酸塩、硝酸塩、ハロゲン化物、シュウ酸塩など高温で酸化物となる化合物または酸化物を用いることができ、これらの金属化合物は従来技術による化合物を用いることがで

(4)

きる。

【0010】

次に、本発明の製造方法における金属化合物の混合物は、工業的に通常用いられている混合方法により各金属化合物を混合して得ることができる。混合する方法は、乾式と湿式のいずれの方法を用いても良い。乾式混合を行う場合に混合に用いる装置は、ボールミル、V型混合機、攪拌装置等の装置を用いることができる。水や有機溶剤を加えて湿式混合をボールミルや攪拌装置を用いて行うこともできる。

【0011】

湿式混合を行った場合には、直接乾燥するかまたは濾過や遠心分離等の方法により固体を液体から分離した後に乾燥する。乾燥温度は20～300℃の範囲が好ましく、さらに好ましくは90～200℃である。直接乾燥させる方法としては、エバポレーションや顆粒化しながら乾燥させるスプレードライを挙げることができる。

【0012】

次に、金属化合物の混合物の焼成においては、最高到達温度は1000℃から1400℃の温度範囲が好ましい。焼成において、1000℃から1400℃の温度範囲に保持する時間は0.5～50時間が好ましい。焼成においては例えば、原料をアルミナボートに充填し、所定のガス雰囲気中で所定の温度で焼成することができる。また必要に応じて、原料に酸化ホウ素、フッ化アルミニウム等の反応促進剤（フラックス）を混合することにより、さらに結晶性が良好で輝度が高いケイ酸塩蛍光体が得られることがある。

【0013】

金属化合物の混合物の中に、金属元素の水酸化物、炭酸塩、硝酸塩、ハロゲン化物、シュウ酸塩など高温で分解し酸化物になりうるものが含まれている場合、前記焼成の前に、400℃から900℃の温度範囲において仮焼することも可能である。

【0014】

上記方法により得られる蛍光体を、ボールミルやジェットミル等を使用して粉碎したり、水等で洗浄することができ、また必要に応じ分級することもできる。得られる蛍光体の結晶性を高めるために、必要に応じて再焼成を行うこともできる。本発明によって得られるケイ酸塩蛍光体の一次粒子の凝集状態は、球状の酸化ケイ素ではない従来のケイ素化合物のみを用いる場合に比べて弱くなり、粉碎等の後の工程が簡略化できる。

【0015】

本発明の製造方法は、金属化合物が、Ca、Sr、Ba、Mg、Eu、Mn、およびZnからなる群から選ばれる一種以上の金属元素の化合物とSiの化合物である場合に好ましく適用でき、本発明の製造方法におけるケイ酸塩蛍光体としては、一般式 $mM^1O \cdot nM^2O \cdot 2SiO_2$ （式中の M^1 はCa、SrおよびBaからなる群より選ばれる1種以上、 M^2 はMgおよびZnからなる群より選ばれる1種以上、mは0.5以上3.5以下、nは0.5以上2.5以下である。）で表される化合物と、付活剤としてEu、Mnからなる群より選ばれる1種以上を含んでなるケイ酸塩蛍光体が好ましい。mが0.5未満の場合、mが3.5を超える場合、nが0.5未満の場合、nが2.5を超える場合のいずれかの場合においては、輝度が高いケイ酸塩蛍光体とはならないおそれがある。

【0016】

例えば、青色蛍光体である $CaMgSi_2O_6:Eu$ を製造するときは、焼成することにより $CaMgSi_2O_6:Eu$ を構成しうるCa、Mg、Euの化合物と球状の酸化ケイ素とを原料とすることができ、さらに酸化ケイ素以外のSiの化合物を加えてもよい。

【0017】

次いで、それらの金属化合物を組成式 $CaMgSi_2O_6:Eu$ で表される化合物からなる蛍光体となるよう、秤量して混合し焼成する。焼成は還元性雰囲気中で、1000℃～1400℃の温度範囲、0.5～40時間の範囲で1回以上焼成することができる。還元性雰囲気を得る方法として、窒素と水素あるいは希ガスと水素の混合雰囲気中で焼成する方法等が挙げられる。また、これらの雰囲気中に水蒸気が含まれていても良い。また、大気中で1000℃以上1400℃以下の温度範囲で焼成した後、還元性雰囲気中で1000

(5)

℃以上1400℃以下の温度範囲で再度焼成することもできる。

【0018】

上記したような本発明の製造方法により得られるケイ酸塩蛍光体は、真空紫外線励起下で高い輝度を有するので、PDPや希ガスランプなどの真空紫外線励起発光素子用に好適である。また、本発明の製造方法により得られるケイ酸塩蛍光体は、真空紫外線のみならず、紫外線、陰極線あるいはX線による励起においても高い輝度を示す。

【0019】

【実施例】

次に、本発明を実施例によりさらに詳しく説明するが、本発明はこれらの実施例に限定されるものではない。

【0020】

実施例1

平均粒径が $0.3\mu\text{m}$ 、BET比表面積が $15.5\text{m}^2/\text{g}$ 、長径/短径比が1.0の球状の酸化ケイ素(SiO_2 、株式会社アドマテックス製)を用いて、炭酸カルシウム(和光純薬工業株式会社製) CaCO_3 、炭酸ストロンチウム(和光純薬工業株式会社製) SrCO_3 、酸化ユーロピウム(信越化学工業株式会社製) Eu_2O_3 、塩基性炭酸マグネシウム(MgCO_3) $4\text{Mg}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$ (和光純薬工業株式会社製)の各原料を $\text{CaCO}_3:\text{SrCO}_3:\text{Eu}_2\text{O}_3:(\text{MgCO}_3)_4\text{Mg}(\text{OH})_2 \cdot 5\text{H}_2\text{O}:\text{SiO}_2$ のモル比が $0.855:0.095:0.025:0.2:2$ になるように配合、混合した後、2体積% H_2 含有Ar雰囲気中で1200℃の温度で2時間保持して焼成した。このようにして、組成式 $\text{Ca}_{0.855}\text{Sr}_{0.095}\text{Eu}_{0.05}\text{MgSi}_2\text{O}_6$ で表される化合物からなる蛍光体を得られた。得られた蛍光体の一次粒子径を走査型電子顕微鏡による蛍光体粒子の写真を用いて計測した結果、すべて $5\mu\text{m}$ 以下であった。この蛍光体に、 6.7Pa ($5 \times 10^{-2}\text{Torr}$)以下の真空槽内で、エキシマ146nmランプ(ウシオ電機社製、H0012型)を用いて紫外線を照射したところ、比較例1の輝度を100とすると、輝度は140であった。

【0021】

実施例2

平均粒径が $0.6\mu\text{m}$ 、BET比表面積が $5.2\text{m}^2/\text{g}$ 、長径/短径比が1.0の球状の酸化ケイ素(SiO_2 、株式会社アドマテックス製)を用いた以外は実施例1と同様にして蛍光体を作製した。この蛍光体に、 6.7Pa ($5 \times 10^{-2}\text{Torr}$)以下の真空槽内で、エキシマ146nmランプ(ウシオ電機社製、H0012型)を用いて紫外線を照射したところ、比較例1の輝度を100とすると、輝度は145であった。

【0022】

実施例3

平均粒径が $0.9\mu\text{m}$ 、BET比表面積が $3.3\text{m}^2/\text{g}$ 、長径/短径比が1.0の球状の酸化ケイ素(SiO_2 、株式会社アドマテックス製)を用いた以外は実施例1と同様にして蛍光体を作製した。この蛍光体に、 6.7Pa ($5 \times 10^{-2}\text{Torr}$)以下の真空槽内で、エキシマ146nmランプ(ウシオ電機社製、H0012型)を用いて紫外線を照射したところ、比較例1の輝度を100とすると、輝度は135であった。

【0023】

実施例4

平均粒径が $1.5\mu\text{m}$ 、BET比表面積が $3.8\text{m}^2/\text{g}$ 、長径/短径比が1.0の球状の酸化ケイ素(SiO_2 、株式会社アドマテックス製)を用いた以外は実施例1と同様にして蛍光体を作製した。この蛍光体に、 6.7Pa ($5 \times 10^{-2}\text{Torr}$)以下の真空槽内で、エキシマ146nmランプ(ウシオ電機社製、H0012型)を用いて紫外線を照射したところ、比較例1の輝度を100とすると、輝度は125であった。

【0024】

比較例1

破砕面を有する不定形の酸化ケイ素(SiO_2 、和光純薬工業株式会社製、カタログ番号

(6)

199-00625)を用いた以外は実施例1と同様にして蛍光体を作製した。実施例1と同様にして輝度を測定した結果、輝度は100であった。

【0025】

【発明の効果】

本発明によれば、輝度が高いケイ酸塩蛍光体を提供することができる。このケイ酸塩蛍光体は真空紫外線励起における輝度が高いので、PDPや希ガスランプなどの真空紫外線励起発光素子に好適な蛍光体であり、本発明の製造方法は工業的に極めて有用である。

(7)

フロントページの続き

Fターム(参考) 4H001 CA02 CA04 CF02 XA08 XA12 XA14 XA20 XA30 XA38 XA56
YA25 YA63

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CLAIMS

[Claim(s)]

[Claim 1]

The manufacture approach of the silicate fluorescent substance characterized by using spherical silicon oxide as one of the metallic compounds in the manufacture approach of the silicate fluorescent substance by calcinating the mixture of metallic compounds.

[Claim 2]

The manufacture approach according to claim 1 that the major axis / minor-axis ratio of spherical silicon oxide are 1.5 or less [1 or more].

[Claim 3]

The manufacture approach according to claim 1 or 2 that the mean particle diameter of spherical silicon oxide is 0.1 micrometers or more 3 micrometers or less.

[Claim 4]

The manufacture approach according to claim 1 to 3 that the BET specific surface area of spherical silicon oxide is below 30m²/g more than 1m²/g.

[Claim 5]

The manufacture approach according to claim 1 to 4 which is the mixture of the compound of the metallic element more than a kind chosen from the group which the mixture of metallic compounds becomes from calcium, Sr, Ba, Mg, Eu, Mn, and Zn, and the compound of Si.

[Claim 6]

The manufacture approach according to claim 1 to 5 that the highest attainment temperature in baking is 1000-degree-C or more temperature requirement 1400 degrees C or less.

[Claim 7]

a silicate fluorescent substance — general formula $mM_1O \cdot nM_2O$ and $2SiO_2$ (M_1 in a formula — calcium —) 3.5 or less [0.5 or more] and n of one or more sorts chosen from the group which consists of Sr and Ba, one or more sorts chosen from the group which M_2 becomes from Mg and Zn, and m are 2.5 or less [0.5 or more]. The manufacture approach according to claim 1 to 6 which is the silicate fluorescent substance which comes to contain one or more sorts chosen from the compound expressed and the group which consists of Eu and Mn as an activator.

[Claim 8]

The silicate fluorescent substance characterized by being obtained by the manufacture approach according to claim 1 to 7.

[Claim 9]

The silicate fluorescent substance according to claim 8 with which a particle 5 micrometers or less is contained for the diameter of a primary particle 80% of the weight or more.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention]

Especially this invention relates to the manufacture approach of the silicate fluorescent substance suitable for vacuum-ultraviolet-radiation excitation light emitting devices, such as a plasma display panel (PDP) and a rare-gas lamp, about the manufacture approach of a silicate fluorescent substance.

[0002]

[Description of the Prior Art]

The silicate fluorescent substance is used for a fluorescent lamp, the Braun tube, the luminous object, the vacuum-ultraviolet-radiation excitation light emitting device, etc. Especially, in vacuum-ultraviolet-radiation excitation light emitting devices, such as PDP, improvement in brightness of a component is desired strongly and the improvement in brightness of the silicate fluorescent substance used for them is called for.

[0003]

The silicate fluorescent substance is manufactured by calcinating the mixture of metallic compounds from the former. silicon oxide uses as metallic compounds used as the source of silicon — having — **** — the conventional manufacture approach — setting — particle shape — the thing of an infinite form — industrial — acquisition — since it was easy and cheap, it was used.

[0004]

[Problem(s) to be Solved by the Invention]

The object of this invention is to offer the fluorescent substance manufactured by the manufacture approach of a silicate fluorescent substance with high brightness, and its manufacture approach.

[0005]

[Means for Solving the Problem]

this invention persons came to complete a header and this invention for the ability of a silicate fluorescent substance with the high brightness of luminescence to be manufactured, when silicon oxide spherical as silicon oxide used as a raw material was used as one of the metallic compounds under this situation in the manufacture approach of the silicate fluorescent substance by calcinating the mixture of metallic compounds, as a result of repeating research wholeheartedly.

[0006]

That is, this invention offers the manufacture approach of the silicate fluorescent substance using spherical silicon oxide as one of the metallic compounds in the manufacture approach of the silicate fluorescent substance by calcinating the mixture of metallic compounds. Moreover, this invention offers said manufacture approach that the major axis / minor-axis ratio of spherical silicon oxide are 1.5 or less [1 or more]. Moreover, the mean particle diameter of silicon oxide with spherical this invention provides with the manufacture approach of a publication said 0.1-micrometer or more either it is [either] 3 micrometers or less. Moreover, this invention provides with the manufacture approach of a publication said either whose BET specific surface area of spherical silicon oxide is below 30m²/g more than 1m²/g. Moreover, this invention provides with the manufacture approach of a publication said either which is the mixture of the compound of the metallic element more than a kind chosen from the group which the mixture of metallic compounds becomes from calcium, Sr, Ba, Mg, Eu, Mn, and Zn, and the compound of Si. Moreover, this invention provides with the manufacture approach of a publication said either whose highest attainment temperature in baking is 1000-degree-C or more temperature requirement 1400 degrees C or less. moreover, this invention — a silicate fluorescent substance — general formula mM1 O-nM2O and 2SiO₂ (M1 in a formula — calcium —) 3.5 or less [0.5 or more] and n of one or more sorts chosen from the group which consists of Sr and Ba, one or more sorts chosen from the group which M2 becomes from Mg and Zn, and m are 2.5 or less [0.5 or more]. Said either which is the silicate fluorescent substance which comes to contain one or more sorts chosen from the compound expressed and the group which consists of Eu and Mn as an activator is provided with the manufacture approach of a publication. Moreover, this invention offers the silicate fluorescent substance obtained by said either by the manufacture approach of a publication. Furthermore, as for this invention, the diameter of a primary particle offers [a particle 5 micrometers or less] the silicate fluorescent substance of said publication contained 80% of the weight or more.

[0007]

[Embodiment of the Invention]

This invention is explained in detail below.

The manufacture approach of the silicate fluorescent substance of this invention uses spherical silicon oxide as one of the metallic compounds in the manufacture approach of a silicate fluorescent substance of being the mixture of metallic compounds and calcinating the mixture which can constitute a silicate fluorescent substance by baking. The compound of the metallic element which is an activator is also contained in said metallic compounds. In the manufacture approach of this invention, silicon oxide is powder which consists of a spherical particle. Although a reason is not clear by using spherical silicon oxide, a fluorescent substance with high brightness is obtained.

[0008]

When it measures using an electron microscope photograph as a particle of silicon oxide, and the biggest thing among the diameters of one primary particle is made into a major axis and the smallest thing is made into a minor axis here, as

for a major axis / minor-axis ratio, 1.5 or less are desirable, and 1.3 or less are still more desirable. In addition, if the particle of silicon oxide is a perfect ball, a major axis / minor-axis ratio will be set to 1. 0.1 micrometers or more 3 micrometers or less are desirable, and is 0.2 micrometers or more 2 micrometers or less more preferably, and the mean particle diameter of silicon oxide is 0.2 micrometers or more 1.7 micrometers or less still more preferably. More than 1m²/g, below 30m²/g is desirable still more desirable, and the BET specific surface area of spherical silicon oxide is below 20m²/g more than 2m²/g. When the major axis / minor-axis ratio of spherical silicon oxide exceed 1.5, mean particle diameter exceeds less than 0.1 micrometers or 3 micrometers and a BET specific surface area exceeds under 1m²/g or 30m²/g, there is a possibility that high brightness may not be obtained.

[0009]

In the manufacture approach of this invention, as compounds other than silicon oxide, the compound or oxide which turns into an oxide at elevated temperatures, such as a hydroxide of a metallic element, a carbonate, a nitrate, a halogenide, and an oxalate, can be used with the metallic compounds contained in the mixture which can constitute a silicate fluorescent substance from mixture of metallic compounds by baking, and these metallic compounds can use the compound by the conventional technique.

[0010]

Next, the mixture of the metallic compounds in the manufacture approach of this invention can mix each metallic compounds by the mixed approach usually used industrially, and can be obtained. Which approach of dry type and wet may be used for the approach of mixing. Equipments, such as a ball mill, a V shaped rotary mixer, and stirring equipment, can be used for the equipment used for mixing when performing dry blending. Water and an organic solvent can be added and wet blending can also be performed using a ball mill or stirring equipment.

[0011]

When wet blending is performed, it dries, after carrying out direct drying or separating a solid-state from a liquid by approaches, such as filtration and centrifugal separation. The range of 20-300 degrees C is desirable still more desirable, and drying temperature is 90-200 degrees C. As an approach of carrying out direct drying, evaporation and the spray dry dried while granulating can be mentioned.

[0012]

Next, in baking of the mixture of metallic compounds, the highest attainment temperature has a 1000 to 1400 degrees C desirable temperature requirement. In baking, the time amount held to a 1000 to 1400 degrees C temperature requirement has 0.5 - 50 desirable hours. In baking, an alumina boat can be filled up with a raw material and it can be calcinated at predetermined temperature in a predetermined gas ambient atmosphere. Moreover, by mixing reaction accelerators (flux), such as boron oxide and aluminum fluoride, in a raw material, crystallinity is still better and a silicate fluorescent substance with high brightness may be obtained if needed.

[0013]

When what decomposes at elevated temperatures, such as a hydroxide of a metallic element, a carbonate, a nitrate, a halogenide, and an oxalate, into the mixture of metallic compounds, and can become an oxide is contained, it is also possible to carry out temporary quenching in a 400 to 900 degrees C temperature requirement before said baking.

[0014]

The fluorescent substance obtained by the above-mentioned approach can be ground using a ball mill, a jet mill, etc., can be washed with water etc., and can also be classified if needed. In order to raise the crystallinity of the fluorescent substance obtained, re-baking can also be performed if needed. The state of aggregation of the primary particle of the silicate fluorescent substance obtained by this invention becomes weak compared with the case where only the conventional silicon compound which is not spherical silicon oxide is used, and can simplify the process after grinding etc.

[0015]

Metallic compounds the manufacture approach of this invention calcium, Sr, Ba, Mg, Eu, When it is the compound of the metallic element more than a kind chosen from the group which consists of Mn and Zn, and the compound of Si, can apply preferably, and as a silicate fluorescent substance in the manufacture approach of this invention General formula $mM1O-nM2O-2SiO_2$ (3.5 [or less / 0.5 or more] and n of one or more sorts chosen from the group which M1 in a formula becomes from calcium, Sr, and Ba, one or more sorts chosen from the group which M2 becomes from Mg and Zn, and m are 2.5 or less [0.5 or more].) The silicate fluorescent substance which comes to contain one or more sorts chosen from the compound expressed and the group which consists of Eu and Mn as an activator is desirable. When m is less than 0.5, m exceeds 3.5 and n is less than 0.5, a possibility that brightness may not serve as a high silicate fluorescent substance is in one in case n exceeds 2.5 of cases.

[0016]

For example, when manufacturing CaMgSi₂O₆:Eu which is a blue fluorescent substance, by calcinating, the compound of calcium, Mg, and Eu which can constitute CaMgSi₂O₆:Eu, and spherical silicon oxide can be used as a raw material, and the compound of Si other than silicon oxide may be added further.

[0017]

Subsequently, weighing capacity is carried out, it mixes and those metallic compounds are calcinated so that it may become the fluorescent substance which consists of a compound expressed with empirical formula CaMgSi₂O₆:Eu. Baking can be calcinated once or more in a reducing atmosphere the temperature requirement of 1000 degrees C - 1400 degrees C, and in 0.5 - 40 hours. As an approach of acquiring a reducing atmosphere, the approach of calcinating in the mixed ambient atmosphere of nitrogen, hydrogen, or rare gas and hydrogen etc. is mentioned. Moreover, the steam may be contained in these ambient atmospheres. Moreover, after calcinating in atmospheric air in 1000-degree-C or more temperature requirement 1400 degrees C or less, it can also calcinate again in a reducing atmosphere in 1000-degree-C or more temperature requirement 1400 degrees C or less.

[0018]

Since the silicate fluorescent substance obtained by the manufacture approach of this invention which was described above has high brightness under vacuum-ultraviolet-radiation excitation, it is suitable for vacuum-ultraviolet-radiation excitation light emitting devices, such as PDP and a rare-gas lamp. Moreover, the silicate fluorescent substance obtained by the manufacture approach of this invention shows high brightness also in excitation not only by vacuum ultraviolet radiation but ultraviolet rays, cathode rays, or an X-ray.

[0019]

[Example]

Next, although an example explains this invention in more detail, this invention is not limited to these examples.

[0020]

Example 1

Mean particle diameter uses [15.5m²/g and a major axis / minor-axis ratio] 0.3 micrometers, and a BET specific surface area uses the spherical silicon oxide (SiO₂, product made from ADOMA, Inc. tex) of 1.0. A calcium carbonate CaCO₃, a strontium carbonate (The Wako Pure Chem Industries make) SrCO₃, oxidation europium (The Wako Pure Chem Industries make) Each raw material of Eu₂O₃ and basic-magnesium-carbonate (MgCO₃) 4Mg(OH)2.5H₂O (the Wako Pure Chem Industries make) CaCO₃:SrCO₃:Eu₂O₃:(MgCO₃)4Mg(OH)2.5H₂O : (Shin-Etsu Chemical Co., Ltd. make) After blending and mixing so that the mole ratio of SiO₂ may be set to 0.855:0.095:0.025:0.2:2, in the 2 volume %H₂ content Ar ambient atmosphere, at the temperature of 1200 degrees C, it held for 2 hours and calcinated. Thus, the fluorescent substance which consists of a compound expressed with empirical formula calcium0.855Sr0.095Eu0.05MgSi₂O₆ was obtained. As a result of measuring the diameter of a primary particle of the obtained fluorescent substance using the photograph of the fluorescent substance particle by the scanning electron microscope, it was 5 micrometers or less altogether. Brightness was 140, when the excimer 146nm lamp (the USHIO, INC. make, H0012 mold) was used, ultraviolet rays were irradiated within the vacuum tub below 6.7Pa (5x10⁻²Torr) at this fluorescent substance and the brightness of the example 1 of a comparison was set to 100.

[0021]

Example 2

Except that mean particle diameter used [5.2m²/g, and a major axis / minor-axis ratio] 0.6 micrometers and the BET specific surface area used the spherical silicon oxide (SiO₂, product made from ADOMA, Inc. tex) of 1.0, the fluorescent substance was produced like the example 1. Brightness was 145, when the excimer 146nm lamp (the USHIO, INC. make, H0012 mold) was used, ultraviolet rays were irradiated within the vacuum tub below 6.7Pa (5x10⁻²Torr) at this fluorescent substance and the brightness of the example 1 of a comparison was set to 100.

[0022]

Example 3

Except that mean particle diameter used [3.3m²/g, and a major axis / minor-axis ratio] 0.9 micrometers and the BET specific surface area used the spherical silicon oxide (SiO₂, product made from ADOMA, Inc. tex) of 1.0, the fluorescent substance was produced like the example 1. Brightness was 135, when the excimer 146nm lamp (the USHIO, INC. make, H0012 mold) was used, ultraviolet rays were irradiated within the vacuum tub below 6.7Pa (5x10⁻²Torr) at this fluorescent substance and the brightness of the example 1 of a comparison was set to 100.

[0023]

Example 4

Except that mean particle diameter used [3.8m²/g, and a major axis / minor-axis ratio] 1.5 micrometers and the BET specific surface area used the spherical silicon oxide (SiO₂, product made from ADOMA, Inc. tex) of 1.0, the fluorescent substance was produced like the example 1. Brightness was 125, when the excimer 146nm lamp (the USHIO, INC. make, H0012 mold) was used, ultraviolet rays were irradiated within the vacuum tub below 6.7Pa (5x10⁻²Torr) at this fluorescent substance and the brightness of the example 1 of a comparison was set to 100.

[0024]

The example 1 of a comparison

The fluorescent substance was produced like the example 1 except having used the silicon oxide (SiO₂, the Wako Pure Chem Industries make, catalog number 199-00625) of an infinite form which has a crushing side. Brightness was 100 as a result of measuring brightness like an example 1.

[0025]

[Effect of the Invention]

According to this invention, a silicate fluorescent substance with high brightness can be offered. Since brightness [in / in this silicate fluorescent substance / vacuum-ultraviolet-radiation excitation] is high, it is a suitable fluorescent substance for vacuum-ultraviolet-radiation excitation light emitting devices, such as PDP and a rare-gas lamp, and the manufacture approach of this invention has it industrially. [very useful]

[Translation done.]

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TECHNICAL FIELD

[Field of the Invention]

Especially this invention relates to the manufacture approach of the silicate fluorescent substance suitable for vacuum-ultraviolet-radiation excitation light emitting devices, such as a plasma display panel (PDP) and a rare-gas lamp, about the manufacture approach of a silicate fluorescent substance.

[0002]

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PRIOR ART

[Description of the Prior Art]

The silicate fluorescent substance is used for a fluorescent lamp, the Braun tube, the luminous object, the vacuum-ultraviolet-radiation excitation light emitting device, etc. Especially, in vacuum-ultraviolet-radiation excitation light emitting devices, such as PDP, improvement in brightness of a component is desired strongly and the improvement in brightness of the silicate fluorescent substance used for them is called for.

[0003]

The silicate fluorescent substance is manufactured by calcinating the mixture of metallic compounds from the former. silicon oxide uses as metallic compounds used as the source of silicon -- having -- *** -- the conventional manufacture approach -- setting -- particle shape -- the thing of an infinite form -- industrial -- acquisition -- since it was easy and cheap, it was used.

[0004]

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EFFECT OF THE INVENTION

[Effect of the Invention]

According to this invention, a silicate fluorescent substance with high brightness can be offered. Since brightness [in / in this silicate fluorescent substance / vacuum-ultraviolet-radiation excitation] is high, it is a suitable fluorescent substance for vacuum-ultraviolet-radiation excitation light emitting devices, such as PDP and a rare-gas lamp, and the manufacture approach of this invention has it industrially. [very useful]

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention]

The object of this invention is to offer the fluorescent substance manufactured by the manufacture approach of a silicate fluorescent substance with high brightness, and its manufacture approach.

[0005]

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MEANS

[Means for Solving the Problem]

this invention persons came to complete a header and this invention for the ability of a silicate fluorescent substance with the high brightness of luminescence to be manufactured, when silicon oxide spherical as silicon oxide used as a raw material was used as one of the metallic compounds under this situation in the manufacture approach of the silicate fluorescent substance by calcinating the mixture of metallic compounds, as a result of repeating research wholeheartedly.

[0006]

That is, this invention offers the manufacture approach of the silicate fluorescent substance using spherical silicon oxide as one of the metallic compounds in the manufacture approach of the silicate fluorescent substance by calcinating the mixture of metallic compounds. Moreover, this invention offers said manufacture approach that the major axis / minor-axis ratio of spherical silicon oxide are 1.5 or less [1 or more]. Moreover, the mean particle diameter of silicon oxide with spherical this invention provides with the manufacture approach of a publication said 0.1-micrometer or more either it is [either] 3 micrometers or less. Moreover, this invention provides with the manufacture approach of a publication said either whose BET specific surface area of spherical silicon oxide is below 30m²/g more than 1m²/g. Moreover, this invention provides with the manufacture approach of a publication said either which is the mixture of the compound of the metallic element more than a kind chosen from the group which the mixture of metallic compounds becomes from calcium, Sr, Ba, Mg, Eu, Mn, and Zn, and the compound of Si. Moreover, this invention provides with the manufacture approach of a publication said either whose highest attainment temperature in baking is 1000-degree-C or more temperature requirement 1400 degrees C or less. moreover, this invention — a silicate fluorescent substance — general formula mM1 O-nM2O and 2SiO₂ (M1 in a formula — calcium —) 3.5 or less [0.5 or more] and n of one or more sorts chosen from the group which consists of Sr and Ba, one or more sorts chosen from the group which M2 becomes from Mg and Zn, and m are 2.5 or less [0.5 or more]. Said either which is the silicate fluorescent substance which comes to contain one or more sorts chosen from the compound expressed and the group which consists of Eu and Mn as an activator is provided with the manufacture approach of a publication. Moreover, this invention offers the silicate fluorescent substance obtained by said either by the manufacture approach of a publication. Furthermore, as for this invention, the diameter of a primary particle offers [a particle 5 micrometers or less] the silicate fluorescent substance of said publication contained 80% of the weight or more.

[0007]

[Embodiment of the Invention]

This invention is explained in detail below.

The manufacture approach of the silicate fluorescent substance of this invention uses spherical silicon oxide as one of the metallic compounds in the manufacture approach of a silicate fluorescent substance of being the mixture of metallic compounds and calcinating the mixture which can constitute a silicate fluorescent substance by baking. The compound of the metallic element which is an activator is also contained in said metallic compounds. In the manufacture approach of this invention, silicon oxide is powder which consists of a spherical particle. Although a reason is not clear by using spherical silicon oxide, a fluorescent substance with high brightness is obtained.

[0008]

When it measures using an electron microscope photograph as a particle of silicon oxide, and the biggest thing among the diameters of one primary particle is made into a major axis and the smallest thing is made into a minor axis here, as for a major axis / minor-axis ratio, 1.5 or less are desirable, and 1.3 or less are still more desirable. In addition, if the particle of silicon oxide is a perfect ball, a major axis / minor-axis ratio will be set to 1. 0.1 micrometers or more 3 micrometers or less are desirable, and is 0.2 micrometers or more 2 micrometers or less more preferably, and the mean particle diameter of silicon oxide is 0.2 micrometers or more 1.7 micrometers or less still more preferably. More than 1m²/g, below 30m²/g is desirable still more desirable, and the BET specific surface area of spherical silicon oxide is below 20m²/g more than 2m²/g. When the major axis / minor-axis ratio of spherical silicon oxide exceed 1.5, mean particle diameter exceeds less than 0.1 micrometers or 3 micrometers and a BET specific surface area exceeds under 1m²/g or 30m²/g, there is a possibility that high brightness may not be obtained.

[0009]

In the manufacture approach of this invention, as compounds other than silicon oxide, the compound or oxide which turns into an oxide at elevated temperatures, such as a hydroxide of a metallic element, a carbonate, a nitrate, a halogenide, and an oxalate, can be used with the metallic compounds contained in the mixture which can constitute a silicate fluorescent substance from mixture of metallic compounds by baking, and these metallic compounds can use the compound by the conventional technique.

[0010]

Next, the mixture of the metallic compounds in the manufacture approach of this invention can mix each metallic compounds by the mixed approach usually used industrially, and can be obtained. Which approach of dry type and wet may be used for the approach of mixing. Equipments, such as a ball mill, a V shaped rotary mixer, and stirring equipment, can be used for the equipment used for mixing when performing dry blending. Water and an organic solvent can be added and wet blending can also be performed using a ball mill or stirring equipment.

[0011]

When wet blending is performed, it dries, after carrying out direct drying or separating a solid-state from a liquid by

approaches, such as filtration and centrifugal separation. The range of 20–300 degrees C is desirable still more desirable, and drying temperature is 90–200 degrees C. As an approach of carrying out direct drying, evaporation and the spray dry dried while granulating can be mentioned.

[0012]

Next, in baking of the mixture of metallic compounds, the highest attainment temperature has a 1000 to 1400 degrees C desirable temperature requirement. In baking, the time amount held to a 1000 to 1400 degrees C temperature requirement has 0.5 – 50 desirable hours. In baking, an alumina boat can be filled up with a raw material and it can be calcinated at predetermined temperature in a predetermined gas ambient atmosphere. Moreover, by mixing reaction accelerators (flux), such as boron oxide and aluminum fluoride, in a raw material, crystallinity is still better and a silicate fluorescent substance with high brightness may be obtained if needed.

[0013]

When what decomposes at elevated temperatures, such as a hydroxide of a metallic element, a carbonate, a nitrate, a halogenide, and an oxalate, into the mixture of metallic compounds, and can become an oxide is contained, it is also possible to carry out temporary quenching in a 400 to 900 degrees C temperature requirement before said baking.

[0014]

The fluorescent substance obtained by the above-mentioned approach can be ground using a ball mill, a jet mill, etc., can be washed with water etc., and can also be classified if needed. In order to raise the crystallinity of the fluorescent substance obtained, re-baking can also be performed if needed. The state of aggregation of the primary particle of the silicate fluorescent substance obtained by this invention becomes weak compared with the case where only the conventional silicon compound which is not spherical silicon oxide is used, and can simplify the process after grinding etc.

[0015]

Metallic compounds the manufacture approach of this invention calcium, Sr, Ba, Mg, Eu, When it is the compound of the metallic element more than a kind chosen from the group which consists of Mn and Zn, and the compound of Si, can apply preferably, and as a silicate fluorescent substance in the manufacture approach of this invention General formula $mM1O-nM2O-2SiO_2$ (3.5 [or less / 0.5 or more] and n of one or more sorts chosen from the group which M1 in a formula becomes from calcium, Sr, and Ba, one or more sorts chosen from the group which M2 becomes from Mg and Zn, and m are 2.5 or less [0.5 or more].) The silicate fluorescent substance which comes to contain one or more sorts chosen from the compound expressed and the group which consists of Eu and Mn as an activator is desirable. When m is less than 0.5, m exceeds 3.5 and n is less than 0.5, a possibility that brightness may not serve as a high silicate fluorescent substance is in one in case n exceeds 2.5 of cases.

[0016]

For example, when manufacturing $CaMgSi_2O_6:Eu$ which is a blue fluorescent substance, by calcinating, the compound of calcium, Mg, and Eu which can constitute $CaMgSi_2O_6:Eu$, and spherical silicon oxide can be used as a raw material, and the compound of Si other than silicon oxide may be added further.

[0017]

Subsequently, weighing capacity is carried out, it mixes and those metallic compounds are calcinated so that it may become the fluorescent substance which consists of a compound expressed with empirical formula $CaMgSi_2O_6:Eu$. Baking can be calcinated once or more in a reducing atmosphere the temperature requirement of 1000 degrees C – 1400 degrees C, and in 0.5 – 40 hours. As an approach of acquiring a reducing atmosphere, the approach of calcinating in the mixed ambient atmosphere of nitrogen, hydrogen, or rare gas and hydrogen etc. is mentioned. Moreover, the steam may be contained in these ambient atmospheres. Moreover, after calcinating in atmospheric air in 1000-degree-C or more temperature requirement 1400 degrees C or less, it can also calcinate again in a reducing atmosphere in 1000-degree-C or more temperature requirement 1400 degrees C or less.

[0018]

Since the silicate fluorescent substance obtained by the manufacture approach of this invention which was described above has high brightness under vacuum-ultraviolet-radiation excitation, it is suitable for vacuum-ultraviolet-radiation excitation light emitting devices, such as PDP and a rare-gas lamp. Moreover, the silicate fluorescent substance obtained by the manufacture approach of this invention shows high brightness also in excitation not only by vacuum ultraviolet radiation but ultraviolet rays, cathode rays, or an X-ray.

[0019]

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EXAMPLE

[Example]

Next, although an example explains this invention in more detail, this invention is not limited to these examples.

[0020]

Example 1

Mean particle diameter uses [15.5m²/g and a major axis / minor-axis ratio] 0.3 micrometers, and a BET specific surface area uses the spherical silicon oxide (SiO₂, product made from ADOMA, Inc. tex) of 1.0. A calcium carbonate CaCO₃, a strontium carbonate (The Wako Pure Chem Industries make) SrCO₃, oxidation europium (The Wako Pure Chem Industries make) Each raw material of Eu₂O₃ and basic-magnesium-carbonate (MgCO₃) 4Mg(OH)2.5H₂O (the Wako Pure Chem Industries make) CaCO₃:SrCO₃:Eu₂O₃:(MgCO₃)4Mg(OH)2.5H₂O : (Shin-Etsu Chemical Co., Ltd. make) After blending and mixing so that the mole ratio of SiO₂ may be set to 0.855:0.095:0.025:0.2:2, in the 2 volume %H₂ content Ar ambient atmosphere, at the temperature of 1200 degrees C, it held for 2 hours and calcinated. Thus, the fluorescent substance which consists of a compound expressed with empirical formula calcium_{0.855}Sr_{0.095}Eu_{0.05}MgSi₂O₆ was obtained. As a result of measuring the diameter of a primary particle of the obtained fluorescent substance using the photograph of the fluorescent substance particle by the scanning electron microscope, it was 5 micrometers or less altogether. Brightness was 140, when the excimer 146nm lamp (the USHIO, INC. make, H0012 mold) was used, ultraviolet rays were irradiated within the vacuum tub below 6.7Pa (5x10⁻²Torr) at this fluorescent substance and the brightness of the example 1 of a comparison was set to 100.

[0021]

Example 2

Except that mean particle diameter used [5.2m²/g, and a major axis / minor-axis ratio] 0.6 micrometers and the BET specific surface area used the spherical silicon oxide (SiO₂, product made from ADOMA, Inc. tex) of 1.0, the fluorescent substance was produced like the example 1. Brightness was 145, when the excimer 146nm lamp (the USHIO, INC. make, H0012 mold) was used, ultraviolet rays were irradiated within the vacuum tub below 6.7Pa (5x10⁻²Torr) at this fluorescent substance and the brightness of the example 1 of a comparison was set to 100.

[0022]

Example 3

Except that mean particle diameter used [3.3m²/g, and a major axis / minor-axis ratio] 0.9 micrometers and the BET specific surface area used the spherical silicon oxide (SiO₂, product made from ADOMA, Inc. tex) of 1.0, the fluorescent substance was produced like the example 1. Brightness was 135, when the excimer 146nm lamp (the USHIO, INC. make, H0012 mold) was used, ultraviolet rays were irradiated within the vacuum tub below 6.7Pa (5x10⁻²Torr) at this fluorescent substance and the brightness of the example 1 of a comparison was set to 100.

[0023]

Example 4

Except that mean particle diameter used [3.8m²/g, and a major axis / minor-axis ratio] 1.5 micrometers and the BET specific surface area used the spherical silicon oxide (SiO₂, product made from ADOMA, Inc. tex) of 1.0, the fluorescent substance was produced like the example 1. Brightness was 125, when the excimer 146nm lamp (the USHIO, INC. make, H0012 mold) was used, ultraviolet rays were irradiated within the vacuum tub below 6.7Pa (5x10⁻²Torr) at this fluorescent substance and the brightness of the example 1 of a comparison was set to 100.

[0024]

The example 1 of a comparison

The fluorescent substance was produced like the example 1 except having used the silicon oxide (SiO₂, the Wako Pure Chem Industries make, catalog number 199-00625) of an infinite form which has a crushing side. Brightness was 100 as a result of measuring brightness like an example 1.

[0025]

[Translation done.]